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TECHNICAL NOTE

No. 1165

TENSILE PROPERTIES OF A SILLIMANTITE REFRACTORY AT ELEVATED TEMPERATURES

By Alfred E. Kunen, Frederick J. Hartwig
and Joseph R. Bressman

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SUMMARY

The tensile strength, the stress-to-rupture characteristics, and the modulus of elasticity of a sillimanite refractory were investigated at various temperatures. The tensile strength varied from a minimum of 8000 pounds per square inch at a temperature of 500° F to a maximum of 19,000 pounds per square inch at 1800° F. The strength at 1950° F was approximately 15,000 pounds per square inch. Heat-treating the tensile specimens for one-half hour at 1800° F increased the tensile strength 35 percent at room temperature and 70 percent at 500° F. No increase in strength was noted at or above 1400° F.

At a temperature of 1600° F, the 100-hour rupture strength was 9600 pounds per square inch and the 1000-hour rupture strength was 8500 pounds per square inch. At 1800° F, the material withstood a stress of 6700 pounds per square inch for 19 hours.

The modulus of elasticity, which was determined only at room temperature, was 20.3×10^6 pounds per square inch and the material was elastic to the point of fracture.

The density of the sillimanite refractory was 0.10 pound per cubic inch or approximately one-third that of high-temperature metal alloys.

INTRODUCTION

The development of the gas turbine and the jet-propulsion engine has instituted an intensive search for materials that will withstand the high stresses existing in rapidly rotating parts at the high gas temperatures necessary for efficient engine operation. High-temperature strength tests on metal alloys indicate that uncooled

turbine blades for aircraft gas turbines may operate satisfactorily at temperatures of approximately 1500° F. With cooling, this temperature is considerably extended but cooling requires an expenditure of power, increases the mechanical complexity of the engine, and may incur a heat loss. A material that will operate at temperatures in excess of 1500° F is therefore desirable. High-temperature refractory materials have been suggested for gas-turbine application (reference 1). Inasmuch as the rotor blades of a turbine are subjected to predominantly tensile stresses, knowledge of the tensile properties of the blade material is essential for the design of a gas turbine. Although some values of tensile strength of refractory materials at room temperature are given in reference 2, a survey of the literature revealed that little had been done to determine their tensile properties at elevated temperatures. Most of the information on the mechanical strength of ceramics was concerned with the values of the moduli of rupture and elasticity determined from transverse bending tests (references 2 to 5). Reference 6, however, presents the results of an investigation of tensile properties at elevated temperatures of several high strength refractory oxide compositions.

Inasmuch as the information available at the time of this investigation was limited, a study of the tensile properties at high temperatures of a promising refractory material was undertaken at the NACA Cleveland laboratory as a basis for the design of a gas turbine with ceramic blades. Equipment for testing ceramic materials to temperatures of 2000° F was developed and a method was devised for evaluating the bending stresses introduced by the test equipment. This paper presents the results of an investigation of the tensile strength of a sillimanite refractory at temperatures from 80° to 1250° F. A few stress-to-rupture tests were conducted at 1400°, 1600°, and 1800° F; the modulus of elasticity was determined at room temperature.

SPECIMENS AND APPARATUS

The test specimen (fig. 1) was 4.25 inches long with a test section approximately 1.25 inches long and a general shape similar to that suggested in reference 7. The specimens were prepared by the Coors Porcelain Company from a sillimanite ore and probably consisted largely of mullite crystals ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and free silica (SiO_2) in the form of cristobalite (reference 8). The fired pieces were ground to finished dimensions by the manufacturer. The original material had a smooth grayish-white surface; but prolonged heating at 1400° F or above, as in the stress-to-rupture tests, caused the surface to change to dark tan. Because the tensile strength of a ceramic

material may be a function of the cross-sectional area of the specimen tested (reference 2), a 0.5-inch-diameter test section was chosen so the area would be about the same as the cross-sectional area of a proposed ceramic turbine blade.

Preliminary experiments were made with gaskets of asbestos paper and woven asbestos cloth placed on the ends of the test specimen to provide a uniform application of the load between the specimen and the grips. A double thickness of woven asbestos cloth (0.05-in. total thickness) was found to be most satisfactory. A photograph of specimens with and without gaskets is shown in figure 2.

Sketches of the grips, which were made of a heat-resistant alloy, are shown in figure 3. The grips consisted of three jaws that formed a split cone matching the ends of the specimen. The jaws were retained by a threaded two-piece cylindrical collar. In order to reduce distortion of the grips during high-temperature testing, they were so designed that the maximum stress was limited to about one-fourth that of the test specimen.

The hydraulic testing machine had a capacity of 120,000 pounds and an error of less than 0.8 percent over the test range of 300 to 4000 pounds. A sketch of the apparatus assembled in the heads of the testing machine is presented in figure 4. All critical surfaces of the tensile-test apparatus were machined to close tolerances. The distance between the heads of the testing machine was made as great as possible to reduce the eccentricity of loading. Optical strain gages with a 1-inch gage length (fig. 5) were used to check the bending stress and to determine the modulus of elasticity. The gage was capable of indicating a strain of 0.000002 inch.

A tubular furnace with a 3-inch inside diameter was used for heating the specimen during testing. The over-all height of the furnace was 15 inches with a resistance wire-wound core 12 inches long. A preliminary survey of the surface-temperature gradient made with three chromel-alumel thermocouples at the ends and at the center of the test section indicated a maximum temperature differential of 5° F at 1800° F. The temperatures for all tests were controlled by a thermocouple at the center of the test section in conjunction with a self-balancing potentiometer and control device. The temperature indicated by this thermocouple was maintained with $\pm 3^\circ$ F of the desired temperature.

A sketch of the apparatus for the stress-to-rupture tests is shown in figure 6. The load on the tensile specimen was applied through the lever arm by weights placed on the end of the arm. The

same type of furnace and furnace control was used in the stress-to-rupture tests as in the tensile-strength tests. The time for failure was recorded by an electric timer that automatically stopped when a specimen failed.

TESTING PROCEDURE

Preliminary examination and alinement check for tensile-strength tests. - The diameter of each specimen was measured in three positions along the length of the test section and in two planes 90° apart at each position. The average of these six readings was taken as the diameter of the specimen. The angles and the concentricity of the specimen were measured to determine whether the specimens were warped or bent. The specimens were X-rayed in two planes 90° apart to locate internal flaws, cracks, or blowholes and each specimen was visually examined for surface pits or chips. No correlation was apparent between tensile strength and flaws found in the preliminary examinations.

The tensile-test apparatus (fig. 4) was first calibrated with a steel specimen; the bending stress due to misalignment inherent in the apparatus was less than 1.5 percent of the average tensile stress up to 25,000 pounds per square inch. This calibration is discussed in the appendix. In all tests of the ceramic specimens, a bending stress less than 2 percent of the average tensile stress was considered satisfactory. The components of the apparatus were marked and the marks alined in the same vertical plane for each test. The apparatus was assembled with a sillimanite specimen in place, a stress of 1000 pounds per square inch was applied, and the whole assembly was tapped axially to seat and aline all the parts. Strain gages were affixed approximately in the plane of maximum bending of the tensile-test apparatus on opposite sides of the specimen test section, as shown in figure 5. Increments of strain were measured up to a stress of 3000 pounds per square inch and the difference in elongation of the two sides was determined. From these strain values, the bending stress could be calculated from equation (2) in the appendix. When the specimen was improperly alined, the stress was reduced to 1000 pounds per square inch and the assembly was again tapped axially. If the specimen was still misaligned, the apparatus was disassembled and a new gasket was fitted to the specimen. When the specimen was satisfactorily alined, the stress was dropped to 1000 pounds per square inch and the strain gages were removed. A chromel-alumel thermocouple was wired to the surface of the specimen and the furnace was lifted and centered over the grips and the specimen.

Tensile-strength tests. - The stress of 1000 pounds per square inch was maintained while the temperature was increased approximately 10° F per minute until the test temperature was reached. After 1 hour at the test temperature, the load on the specimen was increased approximately 4000 pounds per minute from the initial load of 200 pounds (a stress of approximately 1000 lb/sq in.) to the breaking load. The fracture was visually examined to determine the characteristics of the break.

Stress-to-rupture tests. - The grips and loading rods were assembled with a ceramic specimen and placed inside the furnace. The lever arm was supported in an approximately horizontal position by a hydraulic jack and the necessary weights were applied to the end of the lever arm. After the test temperature had been reached and maintained for 1 hour, the load was slowly applied to the specimen. The apparatus was so adjusted that the lever arm was horizontal as indicated by a level. No attempt was made to determine the bending stress in these tests.

RESULTS AND DISCUSSION

Tensile strength. - The specimens were tested in two conditions, "as received" and "heat-treated." The arbitrary heat treatment consisted in heating the specimens in an electric-resistance furnace to 1800° F at an average rate of 15° F per minute, maintaining the temperature one-half hour, and then cooling the specimens at the same average rate as heated.

The results of the tests of specimens as received are shown in table I and of heat-treated specimens in table II. The fractures were examined as recommended in reference 6. A rough fracture is defined as one in which approximately 90 to 100 percent of the surface appears to be granular; this type of break indicates that the entire area was subjected to tension and the entire surface resisted it. A partly rough fracture is one in which 50 to 90 percent of the surface is rough; this type of fracture indicates that part of the specimen was subjected to shear. A smooth fracture is one in which 0 to 50 percent of the surface is rough; this type of fracture occurred in only a few instances. In most tests at a given temperature, specimens with rough fractures had higher values of tensile strength than those with partly rough or smooth breaks. A photograph of a rough and a partly rough break is shown in figure 7.

The variation of the average tensile strength with temperature of the specimens as received and heat-treated is shown in figure 8. The averages of the results obtained with the specimens as received indicate that the tensile strength decreases from 11,000 pounds per square inch at room temperature to 8000 pounds per square inch at 500° F and then increases to a maximum of 19,000 pounds per square inch at 1800° F. At 1950° F, the strength decreases to 15,000 pounds per square inch. Heat-treating the specimens at 1800° F raised the average strength 35 percent at room temperature and 70 percent at 500° F. When the test temperature was increased, the strength gradually decreased from its room-temperature value until it was approximately the same as that of the as received specimens at 1400° F; the strength of the heat-treated specimens was also approximately the same as the as received specimens at 1800° F. The heat treatment given the specimens therefore resulted in an improvement in strength only up to 1400° F. The heat treatment also had a tendency to reduce the spread in the specimen tensile values as shown by the smaller values of percentage average deviation from the mean. (See tables I. and II.)

The unusual variation of tensile strength with temperature and heat treatment may be due to a combination of several factors. Ceramic materials undergo phase changes when heated or cooled with accompanying changes in volume. The chief constituents of the material are probably mullite and cristobalite. (See reference 8 for equilibrium diagram of the system $\text{Al}_2\text{O}_3\text{-SiO}_2$.) When cristobalite is heated, it undergoes a phase inversion from the α to the β form in the temperature range 392° to 500° F with a 5-percent increase in volume (reference 9). Tests on a mullite brick (reference 10) revealed a change in the rate of expansion in the temperature range of 1000° to 1400° F, which may be indicative of a phase change in this constituent. These phase changes may affect the average tensile strength of the material in two ways: The sudden increase in volume of the cristobalite at 500° F may mean that the cristobalite in the specimens expands more rapidly than the mullite particles, thus creating internal stresses that would reduce the test value of tensile strength; and the crystals of the material may be strongest when in one particular phase. In addition, it is known that grinding often introduces high surface stresses in metals and, because the specimens had been ground, residual stresses may have remained in the surface of this material as received. The heat treatment might relieve these stresses and, in addition, might tend to "heal" minute cracks in the surface and thus add to the strength of the material. (See reference 11.)

Modulus of elasticity. - The stress-strain curve determined at room temperature for a specimen as received is plotted in figure 9 and indicates that the material is elastic up to the point of fracture; that is, the material exhibited no plasticity at room temperature. In this test, the load was increased from 1000 to 8000 pounds per square inch and then reduced to 1000 pounds per square inch. Strain measurements indicated no permanent set in the specimen. The load was then raised in increments to obtain the stress-strain relation and the total elongation at 8000 pounds per square inch was the same as previously determined. The curves indicate that a bending stress less than 2.0 percent of the average tensile stress existed. (See appendix for method of calculation.) At room temperature the modulus of elasticity of the material, considered as the average of the slopes of the two curves in figure 9, is 20.3×10^6 pounds per square inch.

Stress-to-rupture strength. - The results of the stress-to-rupture tests are summarized in table III. The material withstood a stress of 6700 pounds per square inch for approximately 19 hours at 1800° F. At 1600° F, the stress-to-rupture strength for 100 hours was 9600 pounds per square inch and the value for 1000 hours, obtained by linear extrapolation on a log-log plot, is approximately 8500 pounds per square inch. The stress-to-rupture test at 1400° F was conducted by first applying a load of 6750 pounds per square inch for approximately 500 hours. The load was increased in increments until the specimen failed after 251 hours at 9600 pounds per square inch. The test at 1400° F was conducted in this manner because of insufficient specimens. For this reason, no check points at 1600° F and 1800° F were obtained.

Strength-density ratio. - The centrifugal stress in a rotating object, such as a turbine blade or rotor, varies directly with its density. The more dense the material, the greater its tensile strength will have to be to support the load at a given speed. The density of most high-temperature metals is approximately 0.30 pound per cubic inch whereas that of the sillimanite specimens was 0.10 pound per cubic inch. For the same strength-density ratio, a metal must have three times the strength of the sillimanite refractory or a tensile strength of 55,000 pounds per square inch at 1300° F and 43,000 pounds per square inch at 1950° F. The equivalent stress-to-rupture strength at 1600° F in a high-temperature metal would be approximately 28,800 pounds per square inch for 100 hours or 25,500 pounds per square inch for 1000 hours. The best high-temperature metals available at present will withstand from 15,000 to 20,000 pounds per square inch for 1000 hours at 1600° F. (See fig. 29 of reference 12, p. 54.)

SUMMARY OF RESULTS

The tensile strength, the stress-to-rupture characteristics, and the modulus of elasticity of a sillimanite refractory were investigated at various temperatures with the following results:

1. The tensile strength of the sillimanite refractory varied from 8000 pounds per square inch at a temperature of 500° F to 19,000 pounds per square inch at 1800° F and decreased to 15,000 pounds per square inch at 1950° F.

2. Heat-treating the material for one-half hour at 1800° F increased the tensile strength 35 percent at room temperature and 70 percent at 500° F. No increase in strength was noted at temperatures greater than 1400° F.

3. The stress-to-rupture strength at 1600° F was 9600 pounds per square inch for 100 hours and 8500 pounds per square inch for 1000 hours. At 1800° F, the material failed after 19 hours at 6700 pounds per square inch.

4. The density of the material is 0.10 pound per cubic inch or roughly one-third that of most high-temperature metals.

5. The modulus of elasticity of the material at room temperature was determined to be 20.3×10^6 pounds per square inch and the material was elastic to the point of fracture.

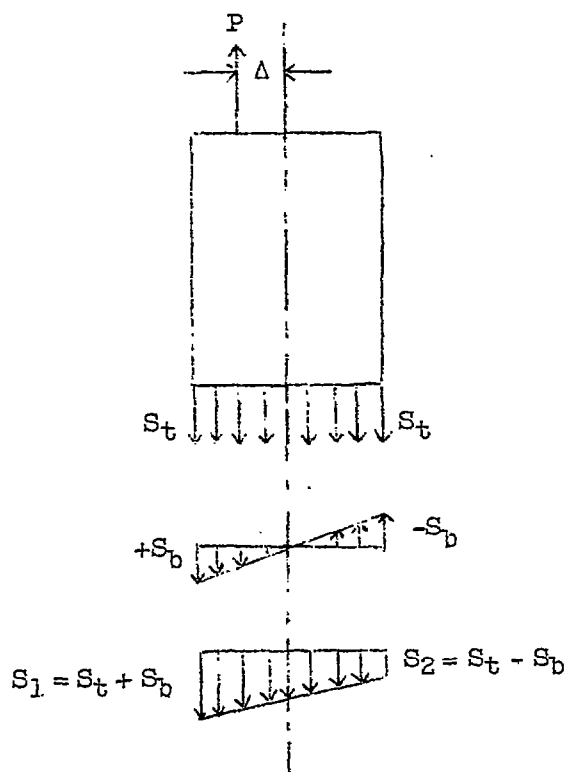
CONCLUSIONS

For the same strength-density ratio as the sillimanite refractory, a high-temperature metal must have an ultimate tensile strength of 55,500 pounds per square inch at 1800° F and 43,000 pounds per square inch at 1950° F. The equivalent stress-to-rupture strength at 1600° F in a high-temperature metal would be 28,800 pounds per square inch for 100 hours or 25,500 pounds per square inch for 1000 hours. These values indicate that sillimanite has sufficient strength to warrant further consideration for application to aircraft gas-turbine blading.

Aircraft Engine Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, July 10, 1946.

APPENDIX - DETERMINATION OF BENDING STRESS IN A TENSILE TEST

Equations for Bending and Modulus of Elasticity



If a load P is applied with an eccentricity Δ to a tensile-test specimen, as shown in the accompanying sketch, the average tensile stress S_t may be superposed on the bending stress S_b , caused by the eccentricity, to give the distribution of fiber stresses as shown. The stresses in the outermost fibers S_1 and S_2 are proportional to the strains ϵ_1 and ϵ_2 in these outermost fibers or

$$E \epsilon_1 = S_t + S_b$$

$$E \epsilon_2 = S_t - S_b$$

Where E is the modulus of elasticity.

Addition of these two equations will eliminate S_b and yield

$$E = \frac{2S_t}{\epsilon_1 + \epsilon_2} \quad (1)$$

Subtraction of the two equations and substitution for E will give

$$S_b = S_t \frac{(\epsilon_1 - \epsilon_2)}{(\epsilon_1 + \epsilon_2)} \quad (2)$$

from which the bending stress may be calculated.

Bending Stress in Apparatus

In order to determine the bending stress and misalignment inherent in the tensile-test apparatus, a specimen of tool steel was machined to the shape of the ceramic specimen. The steel piece was assembled in the grips without a gasket. The strain gages were affixed to the specimen and values of strain on opposite sides were measured with increasing and decreasing load to a maximum stress of 25,000 pounds per square inch. The strain gages were rotated 90° and the procedure was repeated. From these measurements, the maximum bending stress and the position of the plane of maximum bending of the test apparatus were determined for an assumed sinusoidal distribution of stress around the circumference of the specimen test section. This maximum bending stress was found to be about 1.5 percent of the average tensile stress.

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TABLE I - TENSILE TESTS OF SPECIMENS AS RECEIVED

Temperature (°F)	Specimen	Mean diameter (in.)	Load (lb)	Tensile strength (lb/sq in.)	Average tensile strength (lb/sq in.)	Average deviation from mean (percent)	Description of fracture (a)
80	1	0.5039	2445	12,270	11,050	±11.3	PR
	2	.5076	2160	10,670			PR
	3	.5054	2725	13,590			PR
	4	.5016	2130	10,780			PR
	5	.4979	1910	9,810			PR
	6	.4923	1750	9,190			PR
500	7	0.5026	1535	7,730	8,220	±11.1	S
	8	.5050	1905	9,510			PR
	9	.5032	1795	9,030			PR
	10	.4982	1250	6,420			S
	11	.4952	1620	8,410			PR
1000	12	0.5067	2002	9,930	8,620	±12.4	PR
	13	.5025	1305	6,530			S
	14	.5016	1685	8,530			S
	15	.4948	1320	9,460			PR
1400	16	0.5026	2265	11,420	12,000	±10.0	R
	17	.5009	2720	13,800			R
	18	.5005	2125	10,800			PR
1800	19	0.4996	3775	19,260	19,000	±4.7	-----
	20	.5016	3490	17,560			R
	21	.4939	3845	20,070			-----
1950	22	0.4993	2770	14,140	14,700	±3.8	R
	23	.5017	3015	15,250			R

The significance of the letters describing the fractures are:

R Rough, 90-100 percent rough.

PR Partly rough, 50-90 percent rough.

S Smooth, 0-50 percent rough.

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TABLE II - TENSILE TESTS OF HEAT-TREATED SPECIMENS

Temperature (°F)	Specimen	Mean diam- eter (in.)	Load (lb)	Tensile strength (lb/sq in.)	Average tensile strength (lb/sq in.)	Average deviation from mean (percent)	Descrip- tion of fracture (a)
80	24	0.4935	2680	14,020	13,880	±4.8	R
	25	.4991	2870	14,730			R
	26	.4991	2520	12,880			R
500	27	0.5050	2800	13,980	13,580	±6.7	R
	28	.4996	2850	14,540			R
	29	.4950	2350	12,210			R
1000	30	0.5028	2580	13,000	12,270	±3.7	^b PR
	31	.4959	2410	12,480			^c PR
	32	.4982	2170	11,130			^c S
	33	.4987	2400	12,290			R
	34	.4973	2416	12,440			PR
1400	35	0.4998	2350	11,980	11,760	±1.3	R
	36	.4998	2265	11,540			R
1800	37	0.4962	3555	18,380	18,380	-----	R

^aThe significance of the letters describing the fractures are:

R Rough, 90-100 percent rough.

PR Partly rough, 50-90 percent rough.

S Smooth, 0-50 percent rough.

^bPin hole in smooth part of break.

^cBroke in grip section.

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TABLE III - STRESS-TO-RUPTURE TESTS

Temperature (°F)	Specimen	Mean diameter (in.)	Load (lb)	Stress (lb/sq in.)	Time to rupture (hr)
1800	38	0.4963	1896	9400	0.42
	39	.4991	1602	8500	1.17
	40	.4980	1364	6700	19.1
1600	41	0.4992	1957	9600	99.2
	42	.4970	1676	8600	722
1400	43	0.4994	1376	5750	^a 522
			1573	7700	^a 242
			1762	3650	^a 212
			1959	9600	251

^aSpecimen did not fail; but, at the end of this time, the stress was increased.

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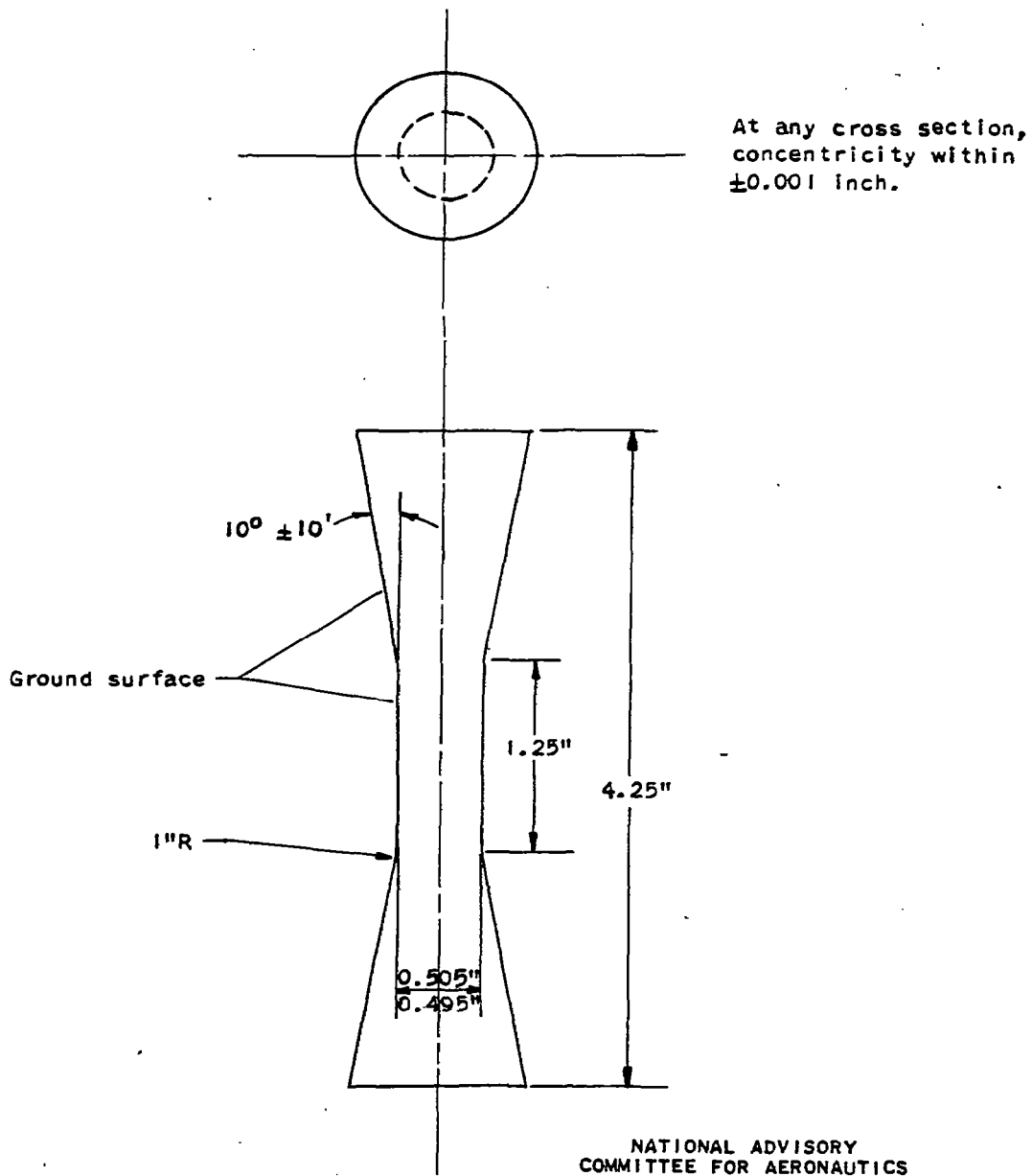


Figure 1. - Sketch of ceramic tensile specimen.

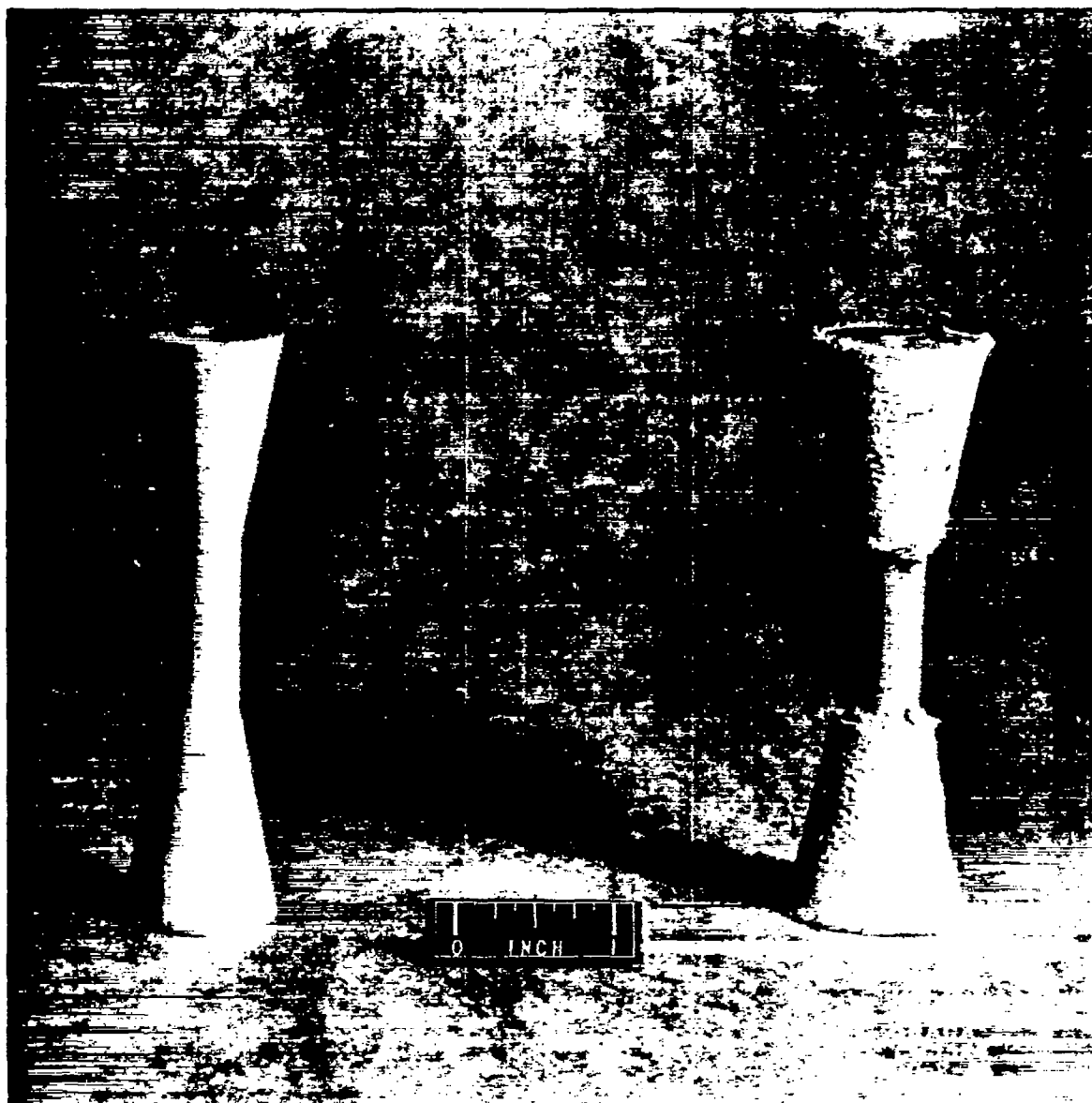
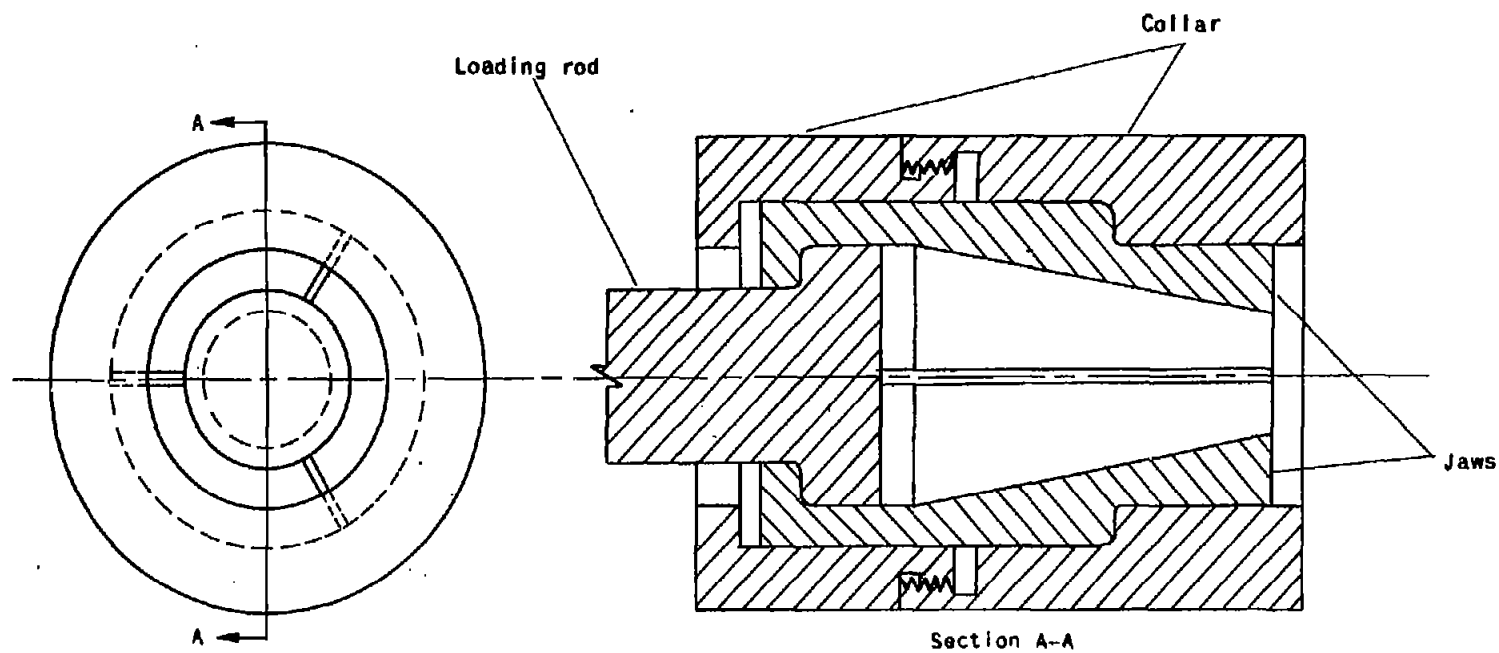


Figure 2. - Sillimanite refractory tensile-test specimens with and without gaskets.



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Figure 3. - Sketch of tensile-test grip.

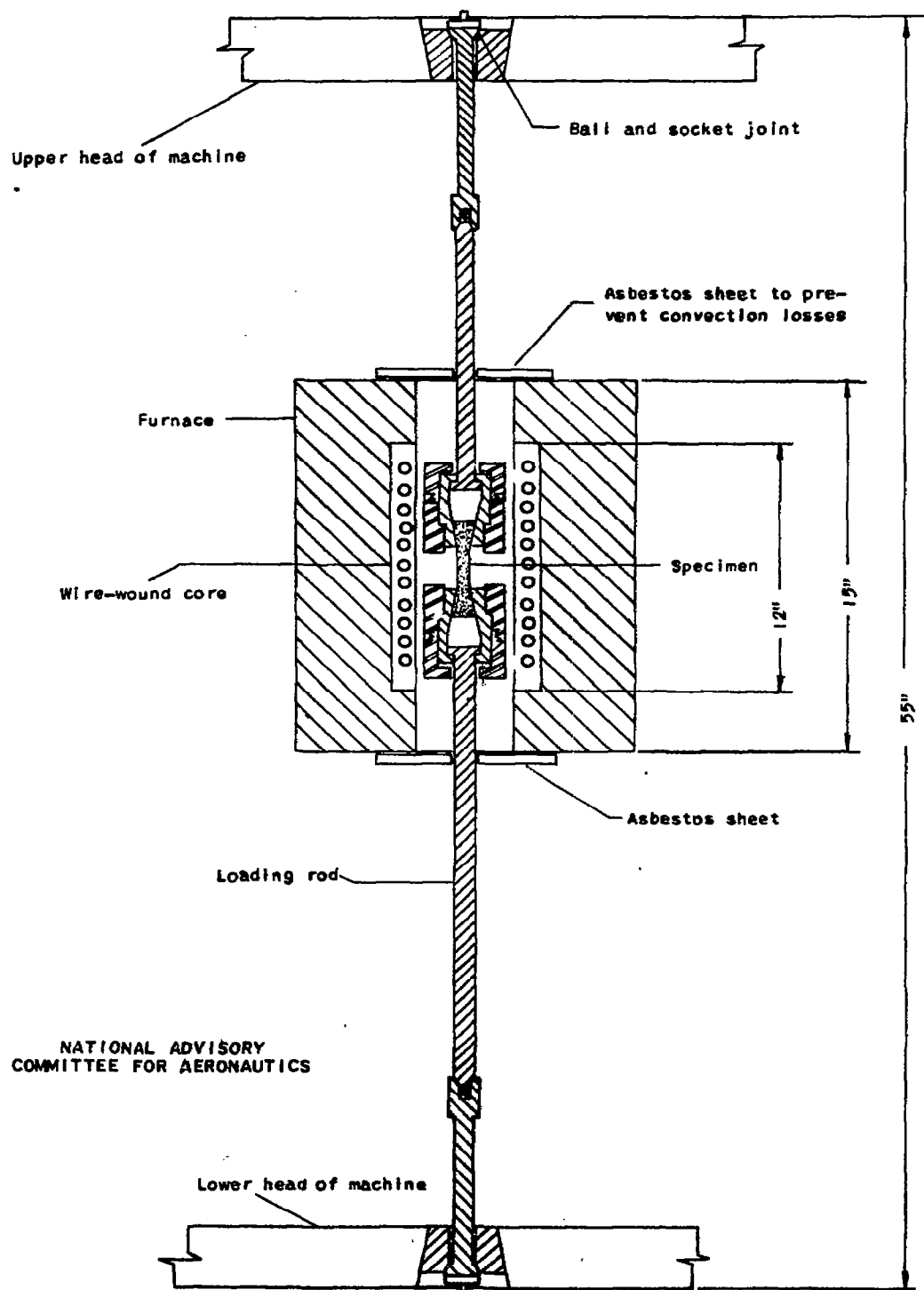


Figure 4. - Sketch of apparatus for tensile tests of ceramic materials.

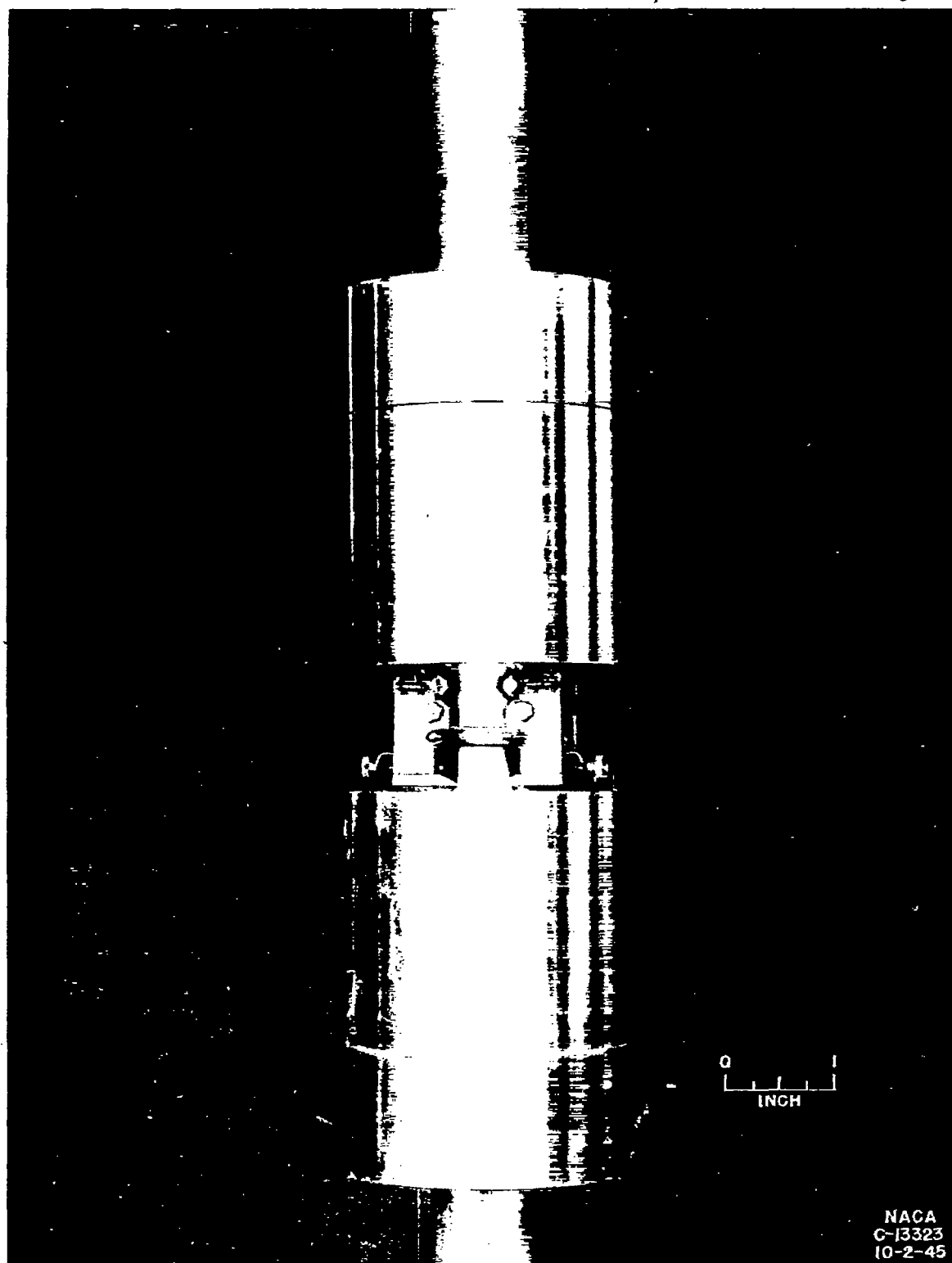


Figure 5. - Optical strain gages affixed to specimen during alignment check.

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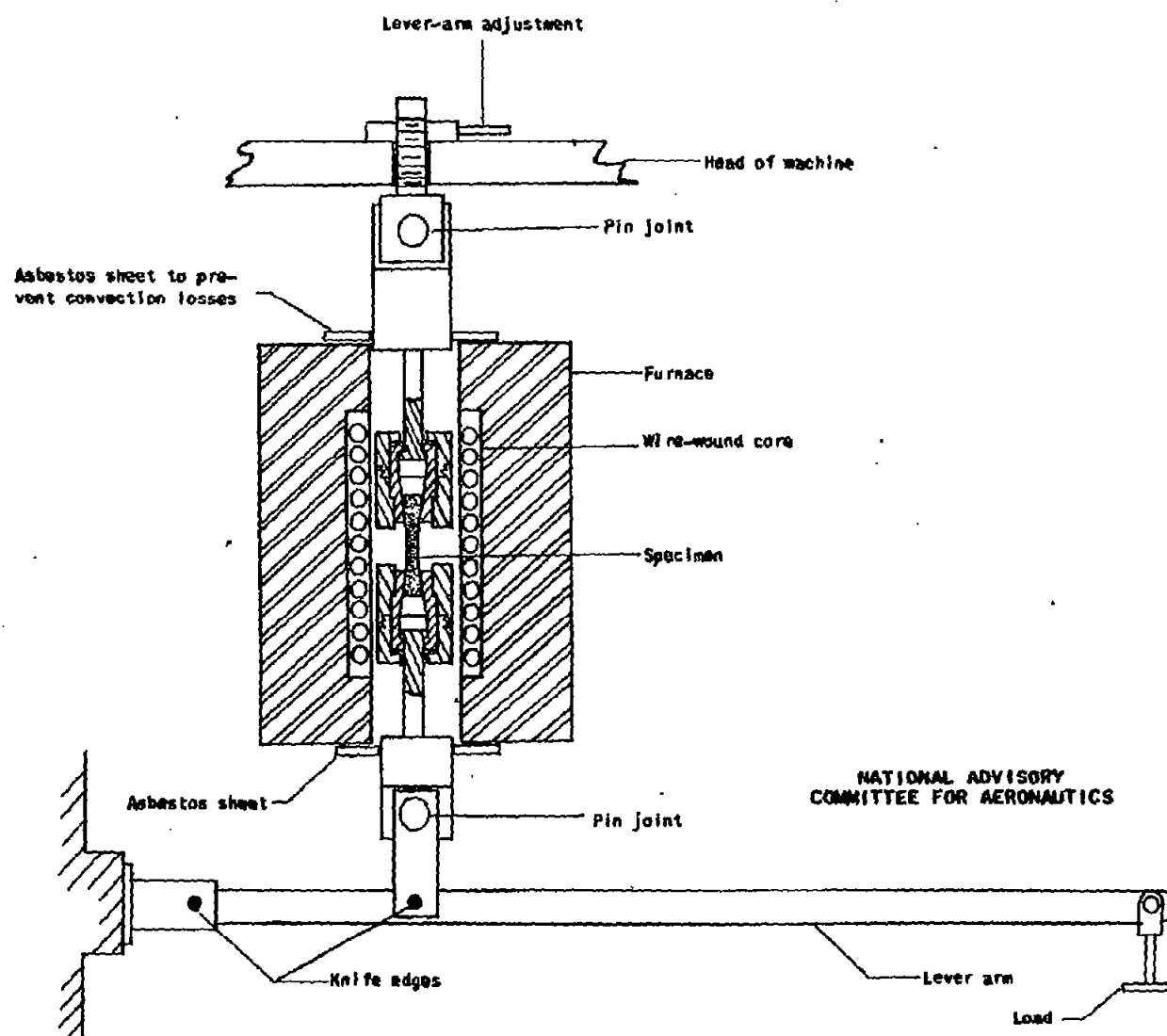


Figure 6. - Sketch of apparatus for stress-to-rupture tests of ceramic materials.



Figure 7. - Typical fractures of sillimanite tensile-test specimens.

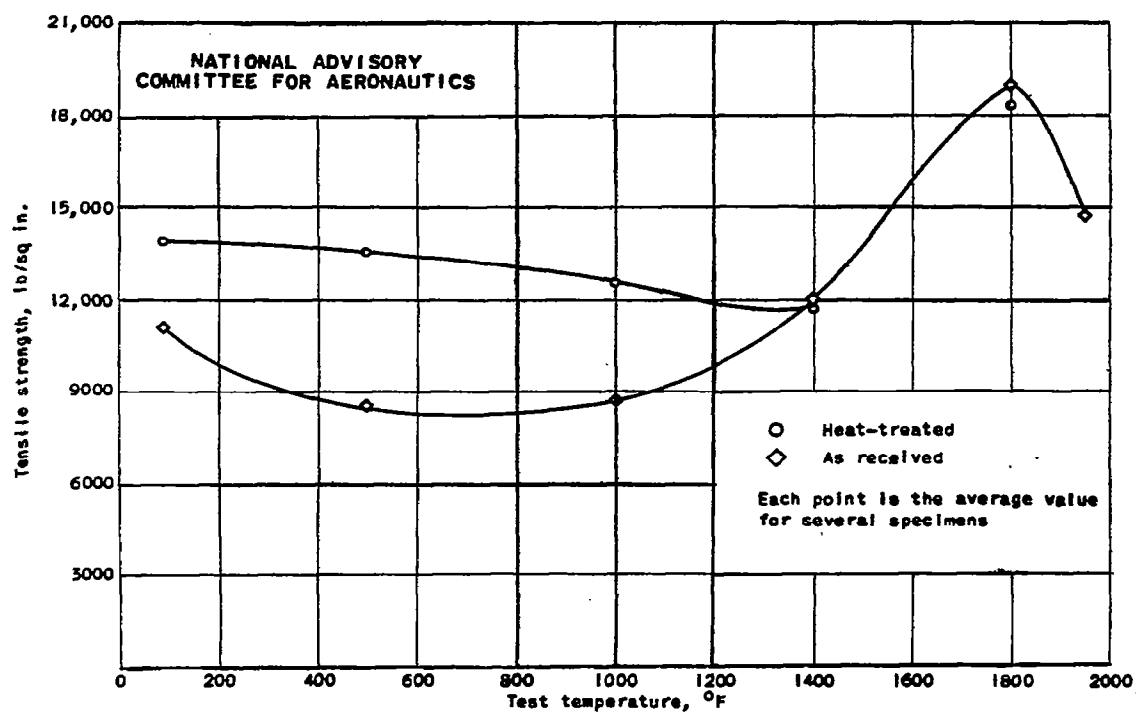


Figure 8. - Variation of tensile strength of sillimanite refractory with temperature.

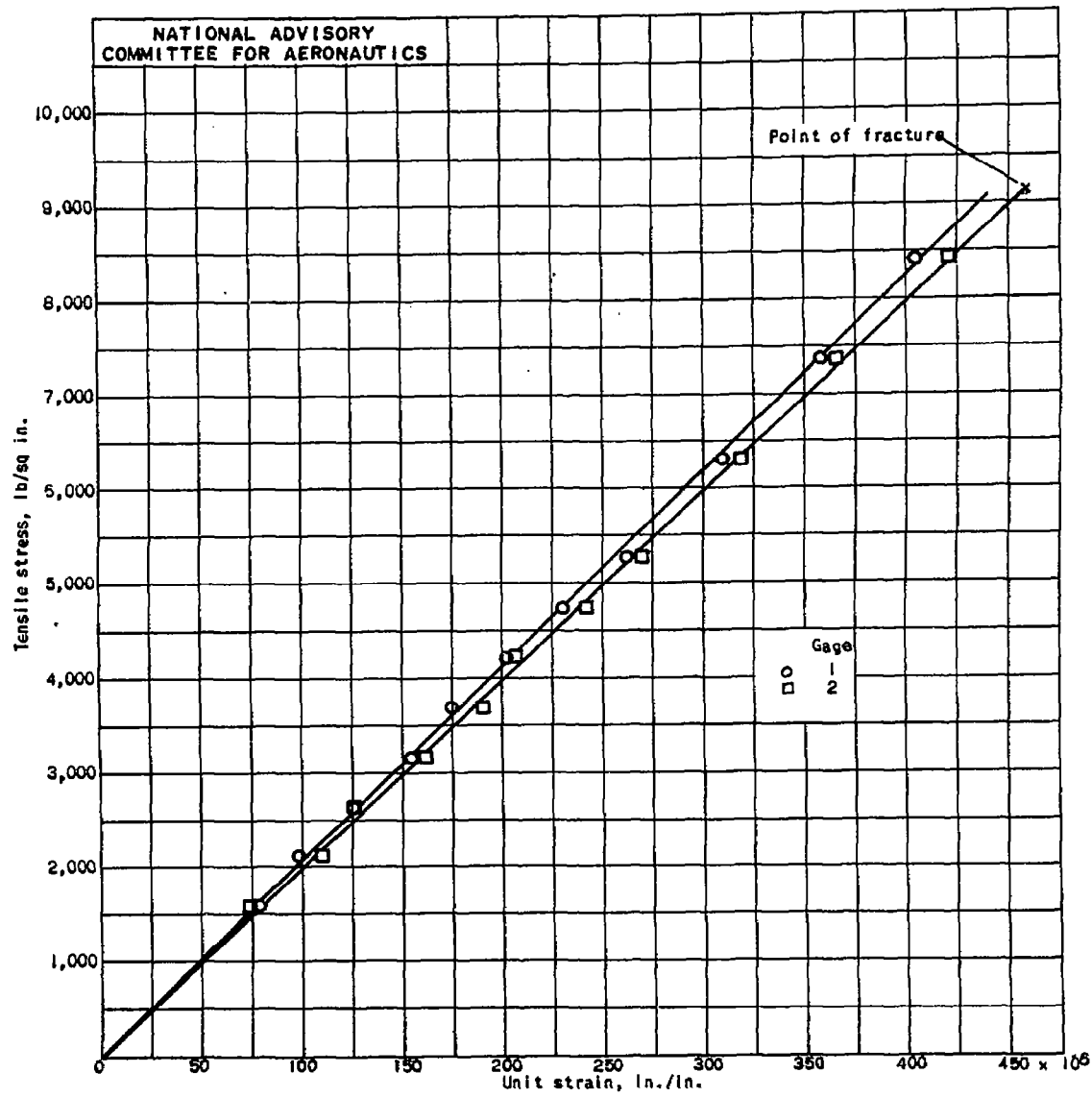


Figure 9. - Stress-strain curve for as received sillimanite refractory at room temperature. Modulus of elasticity, 20.3×10^6 .